

Preparation of HMDS from TMCS at Elevated Temperatures

BALWINDER SINGH GHUMAN

Assistant Professor, Department of Chemical Technology,
Sant Longowal Institute of Engineering & Technology,
Longowal, Distt. Sangrur, Punjab, INDIA.

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ABSTRACT

Hexamethyldisilaxane can be prepared from ammination of Trimethylchlorosilane at elevated temperatures and getting yield up to 68% of TMCS used in the reaction. HMDS is mostly used as a silylating agent with other important uses in chemical syntheses.

Keywords: Trimethylchlorosilane, Hexamethyldisilaxane.

INTRODUCTION

Hexamethyldisilazane, (also known as Bis (trimethylsilyl) amine) is an organosilicon compound with the molecular formula $[(CH_3)_3Si]_2NH$. The molecule is a derivative of ammonia with trimethylsilyl groups in place of two hydrogen atoms. And Si-N bond exists instead of H-N. And if all the H atoms are deleted with Trisilyl groups and we will get Trisilylamine $(H_3Si)_3N$. Which can be prepared by vapor phase reaction of chlorosilane with ammonia. Interesting structural behavior is exists between compounds of $[(CH_3)_2SiNH]_x$ and $[(CH_3)_2SiO]_x$ i.e. dimethylsilaxanes and that of dimethylsiloxanes. The dimethylsiloxanes differs from dimethylsilaxanes only that the siloxane oxygen is replaced by -NH- group however the chemistry differs with the ease

of Hydrolytic cleavage of Si-N bonds. HMDS is a colorless liquid, is a reagent and a precursor to bases that are popular in organic synthesis and organometallic chemistry.

PREPARATION

Bis (trimethylsilyl) amine is prepared by treatment of trimethylsilyl chloride with ammonia in a non reacting solvent. Trimethyl chlorosilane is readily available in the market. Trimethyl chlorosilane is prepared as by product of the reaction of methyl chloride with a silicon-copper alloy. The principal target of this process is dimethyldichlorosilane, but substantial amounts of the trimethyl and monomethyl products are also obtained. HMDS is usually handled using air free

techniques since it hydrolyses slowly in humid air.

PROCEDURE

The reaction vessel is round bottomed flask with a capacity of 1Litre and fitted with reflux condenser. A stirrer is fitted with the flask and a gas inlet tube is also fitted which is lying up to the bottom of the flask. Make sure that it should not touch the stirrer. Generally a large diameter tube is employed to overcome the problem of tube choking with Ammonium chloride. A dry and pure Ammonia gas cylinder is connected to the gas inlet tube with a gas rotameter for feeding of ammonia to the reactor.

About 200gms of sample which contains almost 70% Trimethylchlorosilane and rest solvent is added the reaction vessel and agitation is started. A slow stream of ammonia is started to bubble through the liquid in the vessel and white crystals of ammonium chloride appears. This will continue till the reaction is complete. Do not let the temperature of the reaction mass to go beyond 50°C because TMCS can evaporate at higher temperatures and you will end up in loss of yield. Care should be taken to avoid the loss of TMCS. During the reaction heat is liberated and solvent will continue to reflux in the condenser.

After the addition is complete reflux the solvent for some more time to ensure the reaction completion and to boil off the additional ammonia. Take out the reaction vessel and cool to room temperature. Filter and separate out ammonium chloride by filtration. Wash the ammonium chloride with more quantity of solvent. Solvent is removed by distillation and

Hexamethyldisilaxane is obtained in pure condition. Yield is up to 68-70% of trimethylchlorosilane used.

PROPERTIES OF HMDS

HMDS is colourless liquid, Density $d_4^{20} = 0.7741 \text{ gm/cm}^3$, Refractive index $n_D^{20} = 1.4080(2)$, IUPAC name- Bis(trimethylsilyl)amine is also known as 1,1,1,3,3,3-Hexamethyldisilazane, Molecular formula $\text{C}_6\text{H}_{19}\text{NSi}_2$, Molar mass- 161.39gm/mol, Melting point -78°C, Boiling Point 126°C, Hydrolyses slowly with water (2).

USES

One of the uses of HMDS is as a reagent in condensation reactions of heterocyclic compounds. HMDS can be used to convert alcohols into trimethylsilyl ethers. HMDS can be used to silylate laboratory glassware and make it hydrophobic, or automobile glass, just as rain-X does. In photolithography HMDS is often used as an adhesion promoter for photo resist. In electron microscopy HMDS can be used as an alternative to critical point drying during sample preparation. In pyrolysis gas chromatography mass spectroscopy HMDS is added to the analyze to create silylated diagnostic products during pyrolysis (2)

REFERENCES

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